



3058 Research Drive  
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## **Analytical Report**

**PFOA and PFOS Analysis of Deer Serum Samples by LC/MS/MS**

**MPI Report No. L0019345**

**Revised Report Date: 12/17/09**

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### ***Testing Laboratory***

MPI Research, Inc.  
3058 Research Drive  
State College, PA 16801

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### ***Requester/Project Manager***

Dena Haverland  
Dalton Utilities  
PO BOX 869  
Dalton, GA 30722  
Phone: 706-529-1010

2010 JAN -6 P 1:44

## 1 Introduction

Results are reported for the analysis of PFOS in the 3.5 yr male deer serum sample received at MPI Research from Dalton Utilities. The MPI Research study number assigned to the project is L0019345. Table I lists the target analytes quantitated for the samples.

Table I. Target Analytes for Quantitation

Compound Name	Acronym
Perfluorooctanesulfonate	C8 Sulfonate or PFOS

Note: PFOA results for both the 0.5 yr female and 3.5 yr male are reported in the original report signed on 11/19/09, as well as the PFOS results for the 0.5yr female.

## 2 Sample Receipt

Two samples were received from Dena Haverland at Dalton Utilities for this study. The samples were collected on October 02, 2009. The samples arrived on October 06, 2009 via Fedex and were logged in under MPI Research login number L0019345. The shipment was received frozen on dry ice. The samples were stored frozen at approximately -80°C from receipt until analysis. Chain-of-custody information is presented in Attachment A.

## 3 Methods - Analytical and Preparatory

### 3.1 Serum Sample Preparation

- 3.1.1. Measure 1 mL of serum sample into a 50 mL disposable centrifuge tube and fortify, if appropriate. Add 20 µL of a 50000 ng/mL WIS for a final concentration of 0.5 ng/mL.

Note: The internal standard was spiked at a higher level to allow for post extraction dilutions to be performed.

- 3.1.2 Add water to sample for a final volume of 20 mL. Cap tightly and vortex for ~1 minute.
- 3.1.3 Transfer 1 mL of the sample using a disposable pipette into 15 mL disposable centrifuge tubes. Add 5 mL of ACN and shake for ~20 minutes on a wrist action shaker.
- 3.1.4 Centrifuge tubes at ~3000 rpm for ~ 5 minutes. Carefully decant supernatant into a 50 mL disposable centrifuge tube and add 35 mL of water.
- 3.1.5 Place the unconditioned SPE columns on the vacuum manifold. Condition the SPE columns by passing ~ 10 mL of methanol through the column followed by ~ 5 mL of water. The washes may be pulled through the SPE column using vacuum at a flow rate of ~1 drop/sec or may be allowed to pass through the column unaided. Discard all washes. Do not allow the column to dry.
- 3.1.6 Load the sample onto a conditioned SPE column. Discard the eluate. Any analyte residues will be trapped on the SPE column at this point.

3.1.7 Elute with 2 mL of methanol. Collect 2 mL of elute into a graduated 15 mL centrifuge tube.

Note: Post extraction dilutions were prepared in methanol.

### 3.2 Sample Analysis by LC/MS/MS

In High Pressure Liquid Chromatography (HPLC), an aliquot of extract is injected and passed through a liquid-phase chromatographic column. Based on the affinity of the analyte for the stationary phase in the column relative to the liquid mobile phase, the analyte is retained for a characteristic amount of time. Following HPLC separation, mass spectrometry provides a rapid and accurate means for analyzing a wide range of organic compounds. Molecules are ionized, fragmented, and detected. The ions characteristic of the compounds are observed and quantitated against external calibration standards.

An HP1100 system interfaced to an Applied Biosystems API 4000 LC/MS/MS was used to analyze the sample extracts for quantitation. A gradient elution through a Phenomenex Luna 3 $\mu$  C8(2) Mercury, 20 x 4.0 mm column was used for separation.

The following gradient was performed:

Mobile Phase (A): 2mM Ammonium Acetate in Water  
Mobile Phase (B): Methanol

Time	%A	%B
0.0	90	10
0.5	90	10
2.0	10	90
5.0	10	90
5.1	0	100
6.0	0	100
6.1	90	10
10.0	90	10

The following parameters were used for operation of the mass spectrometer:

Parameter	Setting
Ionization Mode	Electrospray
Polarity	Negative
Transitions Monitored	499 $\rightarrow$ 80 (PFOS) 503 $\rightarrow$ 80 (Internal Std. <sup>13</sup> C PFOS (m+4))
Gas Temperature	450°C

## 4 Analysis by LCMSMS

### 4.1 Calibration

For the serum sample analysis, a 6-point calibration curve was analyzed throughout the analytical sequence for PFOS. The calibration points were prepared at 0.1, 0.2, 0.5, 1.0, 2.0, 5.0 ng/mL (ppb) containing 1.0 ng/mL <sup>13</sup>C-PFOS (m+4).

The ratio of the analyte concentration to the IS concentration versus the ratio of the analyte instrument response (area) to the IS response (area) was plotted for each point. Using linear regression with 1/x weighting, the slope, y-intercept and coefficient of determination ( $r^2$ ) were determined. A calibration curve is acceptable if  $r^2 \geq 0.985$ .

For the results reported here, calibration criteria were met. The calibration curves are included in the raw data in Attachment C.

#### **4.2 Laboratory Control Spikes**

Laboratory control spikes in the analytical set were prepared during each extraction set by adding a known concentration of the analyte to deer serum controls. Laboratory control spikes are used to assess method accuracy. The laboratory control spikes must show recoveries between 70-130% or the data is rejected. For the results reported here, the laboratory control spikes were within the acceptable range. Laboratory control spike recoveries are given in Attachment B.

#### **4.3 Matrix Spikes**

One matrix spike was prepared by adding a known concentration of the target analyte to a sample. Matrix spikes are used to assess method accuracy in the matrix. The matrix spikes should show recoveries between 70-130%. For the results reported here, the matrix spike was within the acceptable range with the exceptions of:

#### **4.4 Laboratory Duplicates**

One sample was prepared in duplicate and analyzed. Duplicate results are given along with the sample results in Attachment B.

### **5 Data Summary**

Please see Attachment B for a detailed listing of the analytical results. For the serum samples the results are reported in parts per billion (ng/mL) on an as-received basis.

### **6 Data/Sample Retention**

Samples are disposed of 60 days after the report is issued unless otherwise specified by the project manager. All electronic data is archived on retrievable media and hard copy reports are stored in data folders maintained by MPI Research. Hardcopy data is stored for a minimum of five years. The client will be notified 30 days prior to the disposal of hardcopy data.



A

## Login

**Login Group: L0019345**

Login #:	19459	Conform COC Sample:	True
Project:	P0005195	Conform COC:	True
Company Name:	Dalton Utilities	Conform Sample:	True
Submitted By:	Dena Haverland	Conform Request:	True
Login Type:	Immediate Receipt of Samples		
Started:	True		
Date Start:	10/27/2009		
Due Date:	11/06/2009		
Login Initiated:	10/27/2009		
Received By:	Ammerman, Mark		
Spread Sample:			
Label:			
MPI SD/PI:	Zhu, Xiang		
Project Title/Type:	PFOA and PFOS Analysis of Serum Samples By LC/MS/MS / ROUTINE		
Login Notes:			

### Packages / Containers

Package	Carton	Date / Condition	Shipper / ID	Temp. Control/Temp.	Direction / Handled By	
K0022041		Received Date: 10/6/09 10:25 Package & Contents Uncompromised	FEDEX 8694 2057 8178	Dry Ice -79.2	RECEIVED Ammerman, Mark	
Container #	Gross Weight	pH	Container Type	Preservative	Mfg. Lot	Mfg. ID
604	3.10 g		2 ml clear plst vial	NONE		
C0457605	3.10 g		2 ml clear plst vial	NONE		
C0457606	3.20 g		2 ml clear plst vial	NONE		
C0457607	3.10 g		2 ml clear plst vial	NONE		
C0457608	3.10 g		2 ml clear plst vial	NONE		
C0457609	3.10 g		2 ml clear plst vial	NONE		
C0457610	3.20 g		2 ml clear plst vial	NONE		
C0457611	3.40 g		2 ml clear plst vial	NONE		
C0457612	3.10 g		2 ml clear plst vial	NONE		
C0457613	3.10 g		2 ml clear plst vial	NONE		
C0457614	3.10 g		2 ml clear plst vial	NONE		
C0457615	3.20 g		2 ml clear plst vial	NONE		
C0457616	3.10 g		2 ml clear plst vial	NONE		
C0457617	3.10 g		2 ml clear plst vial	NONE		
C0457618	3.10 g		2 ml clear plst vial	NONE		
C0457619	3.10 g		2 ml clear plst vial	NONE		
C0457620	3.10 g		2 ml clear plst vial	NONE		
C0457621	3.10 g		2 ml clear plst vial	NONE		
C0457622	3.20 g		2 ml clear plst vial	NONE		
C0457623	3.10 g		2 ml clear plst vial	NONE		



# Login

## Samples

<u>Sample ID</u>	<u>Container</u>	<u>Matrix</u>	<u>System</u>	<u>System Matrix</u>	<u>Sample</u>	<u>Date Sampled</u>	<u>Date Due</u>
L0019345-0001	C0457613	LIQUID	Deer	Serum	Deer #6 0.5 yr female-serum	10/02/2009	11/06/2009
	C0457612						
	C0457611						
	C0457610						
	C0457609						
	C0457608						
	C0457607						
	C0457606						
	C0457605						
	C0457604						
L0019345-0002	C0457617	LIQUID	Deer	Serum	Deer #7 3.5 yr male-serum	10/02/2009	11/06/2009
	C0457623						
	C0457622						
	C0457621						
	C0457620						
	C0457619						
	C0457618						
	C0457616						
	C0457615						
	C0457614						

Login Reviewed By: \_\_\_\_\_



Date/Time: \_\_\_\_\_

12/16/09 1317





# MPI RESEARCH

MPI Research Contact: Daniel Wright

**Send Report To:**  
 Company: Dalton Utilities  
 Address: 1200 VD Parrott JR Parkway, PO Box 869  
 City, State, ZIP: Dalton, GA 30722-0869  
 Attention: Dena Haverland  
 Phone #: 706-529-1010  
 Fax #: 706-529-1271  
 Email: dhaverland@dutil.com  
 Study/Job #: \_\_\_\_\_  
 Signature/Date: \_\_\_\_\_  
 Printed Name: \_\_\_\_\_

## Sample Submittal

Please fax this form before sending samples.

Please send samples to shipping and receiving:  
 3048 Research Drive, State College, PA 16801  
 T: (814) 272-1039 • F: (814) 272-1019

Turnaround time (TAT) requirements:  
 Results Due Date: 30 days  
 Preliminary Results Format: Verbal  Email  Fax  
 Report Due Date: 30 days

Storage Conditions	Safety Information
Room temperature Refrigerator <input checked="" type="radio"/> Freezer Ultra Low freezer Desiccated Lighting required Stability (°C/%RH): _____ Stability time period: _____	Special handling: _____ MSDS attached Controlled substance: _____ HAZARDS: _____ Please fill in the diamond HMIS/NFPA (0-4) if appropriate

Client ID#	Description	Lot/Control #	Amt. Sent/Weight	# of Bottles	Matrix	Date & Time	Tests Requested
1	Deer # 6 0.5 yr female - Serum		10ml	10	deer	10/2/09 1:08am	PFOA/PFOS
2	Deer # 6 0.5 yr female - muscle		as requested	1 bag	deer	10/2/09 2:28am	PFOA/PFOS
3	Deer # 6 0.5 yr female - Liver		Whole	1 bag	deer	10/2/09 2:30am	PFOA/PFOS
4	Deer # 7 3.5 yr Male - Serum		10ml	10	deer	10/2/09 1:45am	PFOA/PFOS
5	Deer # 7 3.5 yr Male - muscle		as requested	1 bag	deer	10/2/09 2:45am	PFOA/PFOS
6	Deer # 7 3.5 yr Male - Liver		Whole	1 bag	deer	10/2/09 2:48am	PFOA/PFOS
7							
8							
9							
10							

PO #: \_\_\_\_\_

Relinquished by	Date	Time	Received by	Date	Time
<u>Daniel Kavanagh</u>	<u>10/5/09</u>	<u>6:30 pm</u>	<u>[Signature]</u>	<u>10/6/09</u>	<u>1025</u>

Notes:

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## TEMPORARY SAMPLE STORAGE FORM

To be completed during ExyLIMS Login

Project #: PS195

Login #: L19345

Initials / Date: MA 10/27/09

One form to be completed for each package

Date / Time Received: 10/06/09 1025

Received By: Mark Ammer

Shipper: FedEx

Shipper Package ID: 8674 2057 8178

Temperature (deg C) / Thermometer ID: -79.2 / D0001775

Temperature Control Method: dry ice - active

Temporary Storage Location: freezer 32

Condition of sample(s):

- Good - Package and contents uncompromised  
 Fair - Package damaged / contents uncompromised  
 Poor - Package and contents compromised

Notes:

# FedEx Express US Airbill

Tracking Number 8694 2057 8178

Form No. 0200

Recipient's Copy

1 From **10/5/09**

Date **10/5/09**

Sender's Name **Darrell Kavanaugh** Phone **706 516-5837**

Company **USDA Wildlife Services/USFS W.I.T.I.S.**

Address **200 Phoenix Road**

City **Athens** State **GA** ZIP **30602**

2 Your Internal Billing Reference

3 To Recipient's Name **Darrell Wright** Phone **814 292-1039**

Company **MOT Research Labs**

Address **3048 Research Drive**

City **State College** State **PA** ZIP **16801**



8694 2057 8178

4a Express Package Service

FedEx Priority Overnight  
Next business morning, Friday  
guaranteed unless otherwise indicated.  
USPS SAT/DAY Delivery is selected.

FedEx Standard Overnight  
Next business afternoon, Saturday Delivery NOT available.

FedEx Express Saver  
Next business day, Saturday Delivery NOT available.

FedEx 2Day  
Second business day, Saturday Delivery NOT available.

FedEx 1Day Freight  
Next business morning, Friday, guaranteed unless otherwise indicated. USPS SAT/DAY Delivery is selected.

FedEx 2Day Freight  
Second business day, Saturday Delivery NOT available.

FedEx 3Day Freight  
Third business day, Saturday Delivery NOT available.

FedEx 4Day Freight  
Fourth business day, Saturday Delivery NOT available.

FedEx 5Day Freight  
Fifth business day, Saturday Delivery NOT available.

FedEx 7Day Freight  
Seventh business day, Saturday Delivery NOT available.

FedEx 9Day Freight  
Ninth business day, Saturday Delivery NOT available.

FedEx 12Day Freight  
Twelfth business day, Saturday Delivery NOT available.

FedEx 15Day Freight  
Fifteenth business day, Saturday Delivery NOT available.

FedEx 20Day Freight  
Twentieth business day, Saturday Delivery NOT available.

FedEx 30Day Freight  
Thirtieth business day, Saturday Delivery NOT available.

FedEx 45Day Freight  
Forty-fifth business day, Saturday Delivery NOT available.

4b Express Freight Service

FedEx 1Day Freight  
Next business morning, Friday, guaranteed unless otherwise indicated. USPS SAT/DAY Delivery is selected.

FedEx 2Day Freight  
Second business day, Saturday Delivery NOT available.

FedEx 3Day Freight  
Third business day, Saturday Delivery NOT available.

FedEx 4Day Freight  
Fourth business day, Saturday Delivery NOT available.

FedEx 5Day Freight  
Fifth business day, Saturday Delivery NOT available.

FedEx 7Day Freight  
Seventh business day, Saturday Delivery NOT available.

FedEx 9Day Freight  
Ninth business day, Saturday Delivery NOT available.

FedEx 12Day Freight  
Twelfth business day, Saturday Delivery NOT available.

FedEx 15Day Freight  
Fifteenth business day, Saturday Delivery NOT available.

FedEx 20Day Freight  
Twentieth business day, Saturday Delivery NOT available.

FedEx 30Day Freight  
Thirtieth business day, Saturday Delivery NOT available.

FedEx 45Day Freight  
Forty-fifth business day, Saturday Delivery NOT available.

FedEx 60Day Freight  
Sixtieth business day, Saturday Delivery NOT available.

FedEx 75Day Freight  
Seventy-fifth business day, Saturday Delivery NOT available.

FedEx 90Day Freight  
Ninety business day, Saturday Delivery NOT available.

FedEx 105Day Freight  
One hundred five business days, Saturday Delivery NOT available.

5 Packaging

FedEx Pak\*  
Includes FedEx Small Pak, FedEx Large Pak, and FedEx Sure Pak.

FedEx Box

FedEx Tube

Other

Special Handling

SATURDAY Delivery

HOLD Weekend at FedEx Location

HOLD Secondary at FedEx Location

HOLD Tertiary at FedEx Location

HOLD Quaternary at FedEx Location

HOLD Quinary at FedEx Location

HOLD Senary at FedEx Location

HOLD Septenary at FedEx Location

HOLD Octenary at FedEx Location

HOLD Nonary at FedEx Location

HOLD Decenary at FedEx Location

6 Special Handling

SATURDAY Delivery

HOLD Weekend at FedEx Location

HOLD Secondary at FedEx Location

HOLD Tertiary at FedEx Location

HOLD Quaternary at FedEx Location

HOLD Quinary at FedEx Location

HOLD Senary at FedEx Location

HOLD Septenary at FedEx Location

HOLD Octenary at FedEx Location

HOLD Nonary at FedEx Location

HOLD Decenary at FedEx Location

HOLD Undecenary at FedEx Location

HOLD Duodecenary at FedEx Location

HOLD Tredecenary at FedEx Location

HOLD Quattuordecenary at FedEx Location

HOLD Quindecenary at FedEx Location

7 Payment Bill to

Sender

Recipient

Third Party

Credit Card

Cash/Check

Other

Dry Ice

Cargo Aircraft Only

Other Restrictions

8 Residential Delivery Signature Options

No Signature Required

Direct Signature

Indirect Signature

Signature Required

9 Total Packages

Total Weight

Total Declared Value

10 Total Packages

Total Weight

Total Declared Value

11 Total Packages

Total Weight

Total Declared Value

12 Total Packages

Total Weight

Total Declared Value

13 Total Packages

Total Weight

Total Declared Value

14 Total Packages

Total Weight

Total Declared Value

15 Total Packages

Total Weight

Total Declared Value

16 Total Packages

Total Weight

Total Declared Value

17 Total Packages

Total Weight

Total Declared Value

**B**



3058 Research Drive  
State College, Pennsylvania 16801 USA  
Telephone: 814.272.1039  
Fax: 814.272.1019

# Analytical Report

## Summary of Fluorochemical Residues in Serum Samples

Sample ID	PFOS Perfluorooctanesulfonate
	Analyte Found (ng/mL, ppb)
Deer # 7 3.5 yr male-serum	1140
Deer # 7 3.5 yr male-serum*	1120

\*Laboratory Duplicate

ND = Not detected = Response is below the LOD of 1.0 ng/mL (ppb).

NQ = Not quantifiable = Response is between the LOD and the LOQ of 10 ng/mL (ppb).



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## Recovery Summary of Fluorochemical Residues in Serum Samples

Sample Description	PFOS			
	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
LCS A (Data set 120709A) 1000 ng/mL	1000	ND	1050	105
LCS B (Data set 120709A) 1000 ng/mL	1000	ND	1100	110
Deer # 7 3.5 yr male-serum (L19345-2 Spk C, 1000 ng/mL Lab Spike)	1000	1140	2130	99

ND = Not detected = Response is below the LOD of 1.0 ng/mL.

NQ = Not quantifiable = Response is between the LOD and the LOQ of 10 ng/mL.

C

RAW DATA REPORT

Sponsor Study No: NA  
 MPI Study No: L19345  
 Analyte: PFOS  
 Ions Monitored: 499 -> 80  
 Site: NA

Limit of Quantitation: 10 ng/mL  
 Injection Volume: 15 µL  
 Matrix: Deer Serum

Set No: 120709A  
 Analyst: Mark Neeley  
 Instrument Type: LC/MS/MS Unit # 9  
 Extraction Date: 12/07/09  
 Analyzed on: 12/07/09

MPI Research ID	Sponsor ID	Sample Code	Sample Index No.	Internal				Peak Area	Internal		Recovery (%)
				Std. Conc. (ng/mL)	Std. Conc. (ng/mL)	Aliquot Factor (AF)	Dilution Factor (DF)		Standard Peak Area	Analyte Found (ng/mL)	
SS33618	-	CS	1	0.100	1.0	-	-	32357	288516	-	
SS33617	-	CS	2	0.200	1.0	-	-	57528	266317	-	
SS33616	-	CS	3	0.500	1.0	-	-	136098	262877	-	
SS33615	-	CS	4	1.00	1.0	-	-	283539	268037	-	
SS33614	-	CS	5	2.00	1.0	-	-	571538	269425	-	
SS33613	-	CS	6	5.00	1.0	-	-	1342568	263402	-	
Methanol Wash	-	W	7	-	-	-	-	1015	25	-	
Methanol Wash	-	W	8	-	-	-	-	1046	85	-	
Control	MC4311 Deer Serum Control	C	9	-	0.5	40	1	1056	73370	ND	
LCS A	MC4311 Deer Serum Spike A, 1000 ng/mL	LCS	10	-	2.5	40	10	479561	440582	1046	
LCS B	MC4311 Deer Serum Spike B, 1000 ng/mL	LCS	11	-	2.5	40	10	469874	412103	1097	
L19345-2 Spk C	Deer # 7 3.5 yr male-serum Spike C, 1000 ng/ml	LF	12	-	2.5	40	10	962941	435982	2134	
L19345-2	Deer # 7 3.5 yr male-serum	S	13	-	2.5	40	10	493540	418410	1135	
L19345-2 Dup	Deer # 7 3.5 yr male-serum Duplicate	S	14	-	2.5	40	10	440374	379350	1117	
SS33615	CCV, 1.0 ng/mL	CCV	15	1.00	1.0	1	1	286632	268817	1.02	

Analyte Found (ng/mL) = (((analyte peak area/IS peak area) - intercept) / slope) x IS conc. x AF x DF

Standard Curve : Linear (1/x weighted)

Recovery (%) =  $\frac{[\text{Analyte found (ng/mL)} - \text{Analyte found in control (ng/mL)}]}{\text{amount Analyte added (ng/mL)}} \times 100$

Intercept = 0.0106

Slope = 1.03

Coef. Of Det. = 0.9996

CS = Calibration standard  
 CCV = Continuing Calibration Verification  
 C = Control sample  
 S = Sample

LF = Lab fortified sample  
 FF = Field fortified sample  
 LCS = Laboratory Control Spike

W = Methanol Wash  
 ND = Not detected = Response between 0 and LOD  
 NQ = Not quantifiable = Response between LOD and LOQ

Spreadsheet prepared by: MN 112-8-09





*Mix 12-7-09*

Object: \\sc1wp5556\mdrive\PE SCIEX DATA\Projects\P5195 Batch:09\_120709A Tab:Sample Set:SET1 AcqMethod:P5195\_102909.d  
Sample

	Sample Name	Sample ID	Vial Position	Data File
1	SS33618	Calibration Standard, 0.1 ng/mL	11	09_120709A\120709A
	SS33617	Calibration Standard, 0.2 ng/mL	12	09_120709A\120709A
3	SS33616	Calibration Standard, 0.5 ng/mL	13	09_120709A\120709A
4	SS33615	Calibration Standard, 1.0 ng/mL	14	09_120709A\120709A
	SS33614	Calibration Standard, 2.0 ng/mL	15	09_120709A\120709A
6	SS33613	Calibration Standard, 5.0 ng/mL	16	09_120709A\120709A
7	Methanol Wash	Methanol Wash	91	09_120709A\120709A
	Methanol Wash	Methanol Wash	91	09_120709A\120709A
9	Control	MC4311 Deer Serum Control	21	09_120709A\120709A
10	LCS A	MC4311 Deer Serum Spike A, 1000 ng/mL, DF=50 <sub>y</sub>	22	09_120709A\120709A
11	LCS B	MC4311 Deer Serum Spike B, 1000 ng/mL, DF=50 <sub>y</sub>	23	09_120709A\120709A
12	L19345-2 Spk C	Deer # 7 3.5 yr male-serum Spike C, 1000 ng/mL, DF=50 <sub>y</sub>	24	09_120709A\120709A
13	L19345-2	Deer # 7 3.5 yr male-serum, DF=50 <sub>y</sub>	25	09_120709A\120709A
14	L19345-2 Dup	Deer # 7 3.5 yr male-serum Duplicate, DF=50 <sub>x</sub>	26	09_120709A\120709A
15	SS33615	CCV, 1.0 ng/mL	14	09_120709A\120709A

\* 10 (10) mix 12-8-09

## LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Protocol No: NA

MPI Study No: L19345

**Instrument:** AB API 4000 Biomolecular Mass Analyzer, (LC/MS/MS #9)  
 SCIEX Turbo Ion Spray Liquid Introduction Interface  
 Turbo Ion spray temperature = 450 °C

**Computer:** Dell OptiPlex GX 110

**Software:** PE Sciex Analyst 1.4

**HPLC Equipment:** Hewlett Packard (HP) Series 1100  
                           HP Quat Pump                           HP Vacuum Degasser  
                           HP Autosampler                        HP Column Oven

**HPLC Column:** Phenomenex Luna C8 (2) Mercury, 2cm x 4mm, 3 µm (ExyLIMS ID: MA0052622)

**Column Temperature:** 35°C

**Mobile Phase (A):** 2 mM Ammonium Acetate in Water (ExyLIMS ID: SL0045925)

**Mobile Phase (B):** Methanol (ExyLIMS ID: RE0047880)

**Injected Volume:** 15 µL

<u>Time (min)</u>	<u>% A</u>	<u>% B</u>	<u>Flow Rate</u> <u>(µL/min)</u>
0.0	90	10	750
0.5	90	10	750
2.0	10	90	750
5.0	10	90	750
5.1	0	100	750
6.0	0	100	750
6.1	90	10	750
10.0	90	10	750

Ions monitored:

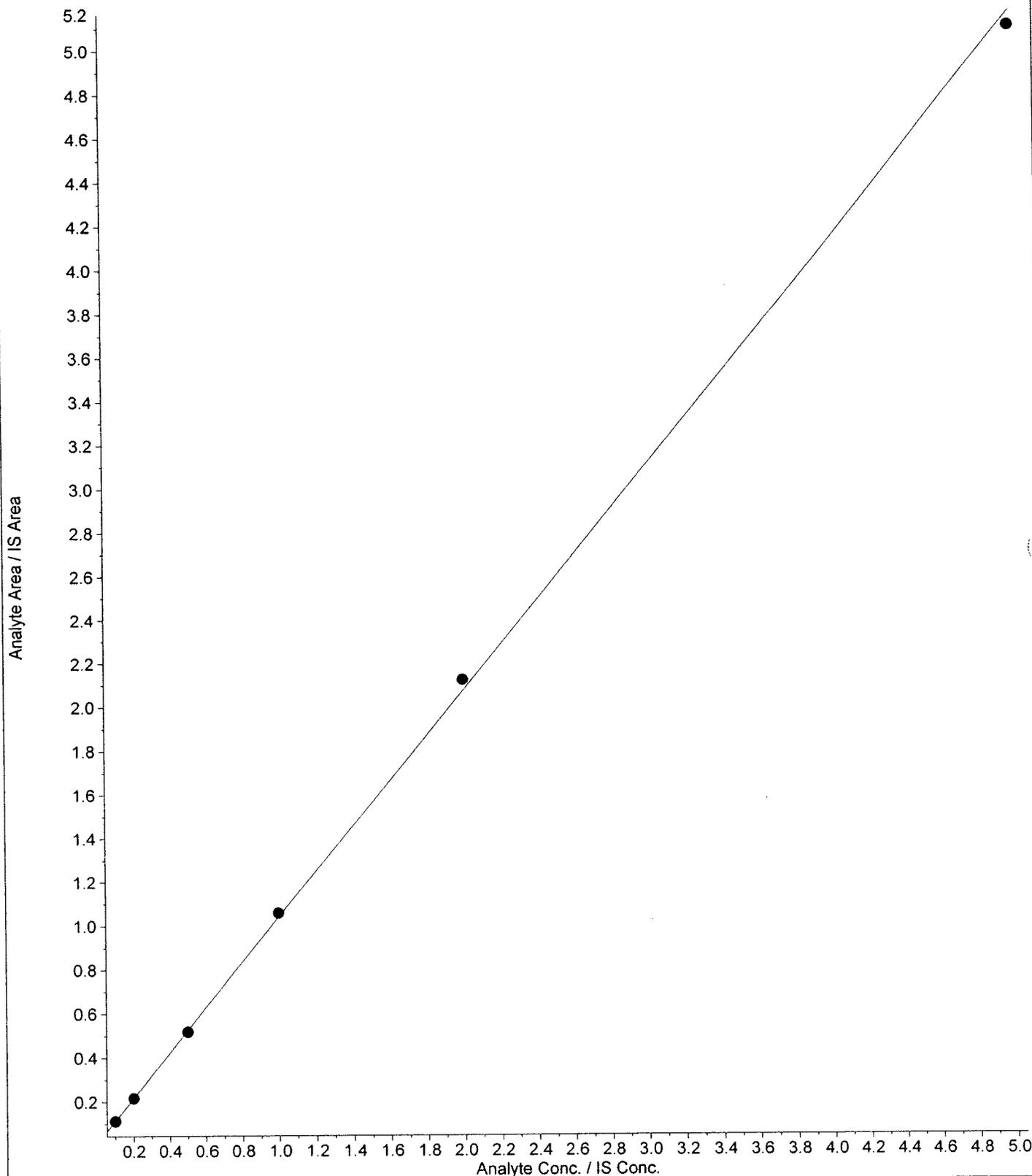
<u>Analyte</u>	<u>Parent ion</u>	<u>Daughter ion(s)</u>	<u>Dwell (secs)</u>
PFOS	499	80	0.200
<sup>13</sup> C PFOS (m+4)	503	80	0.200
Internal Standard			

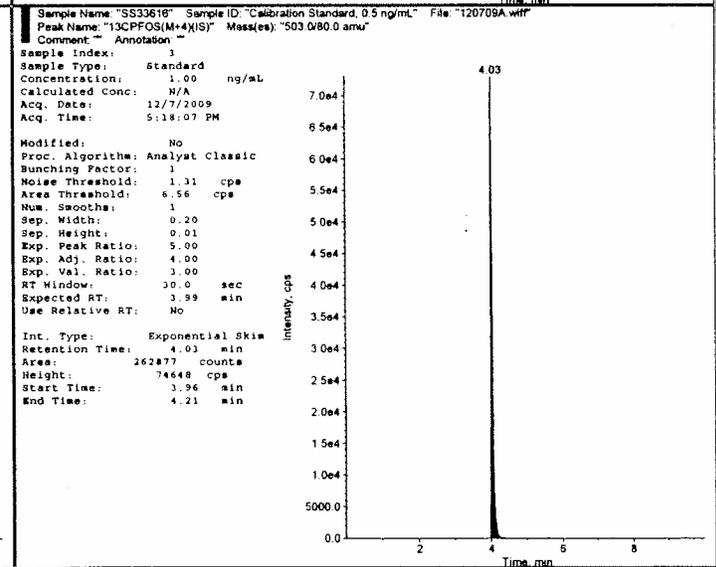
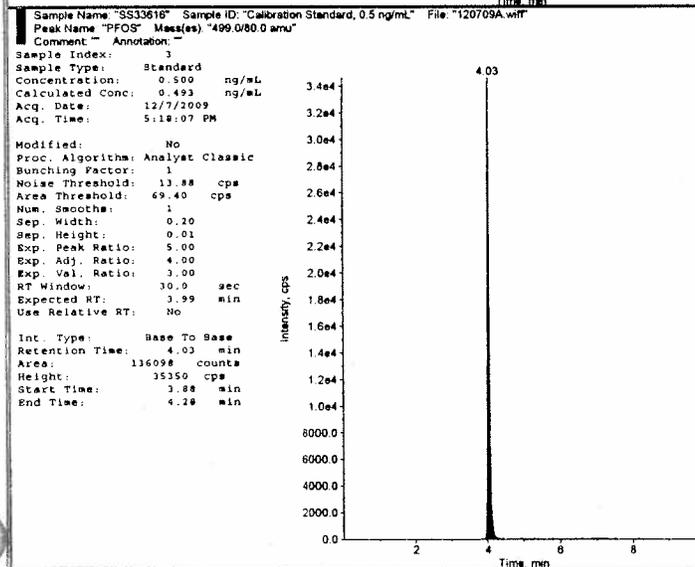
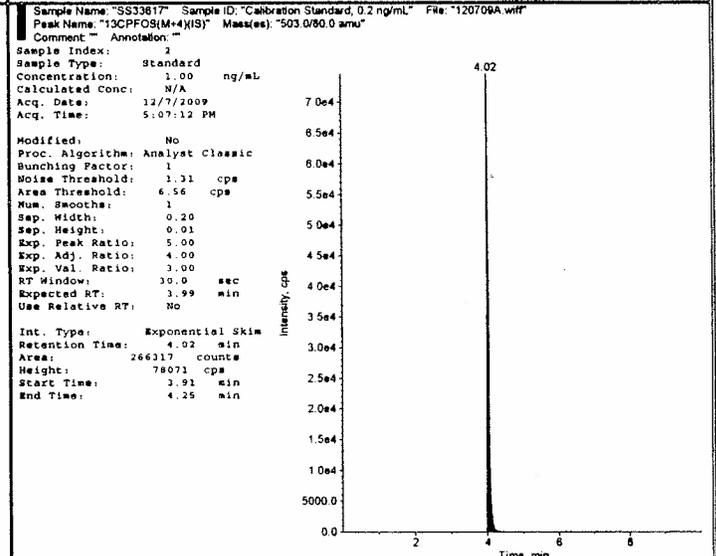
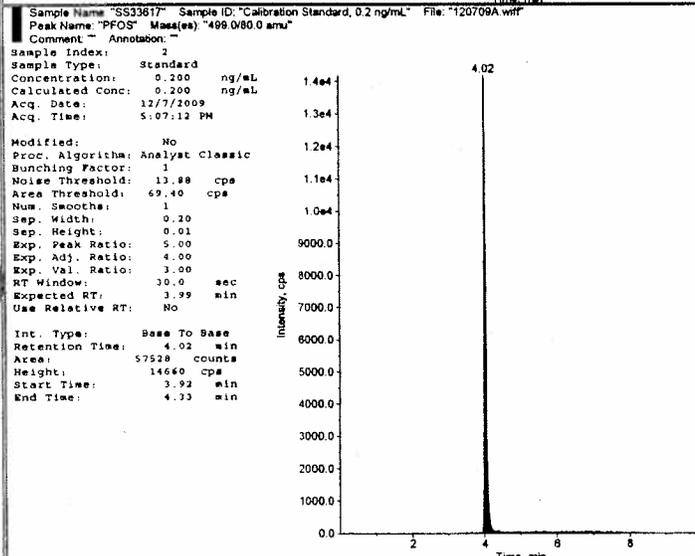
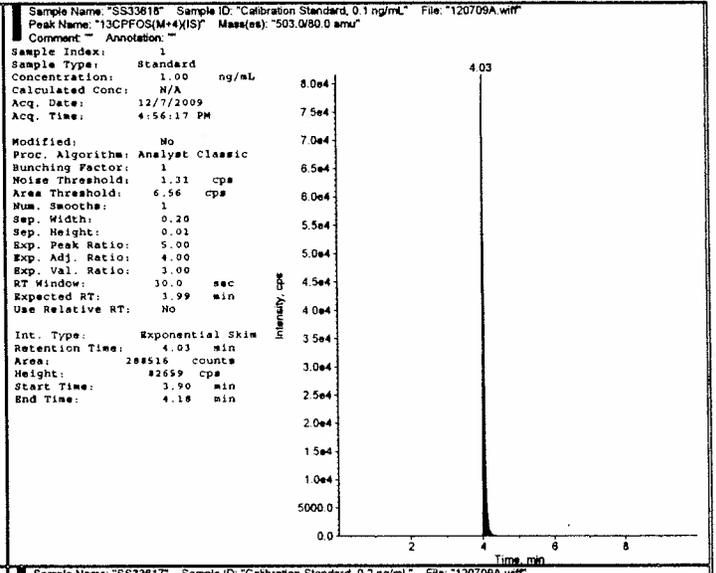
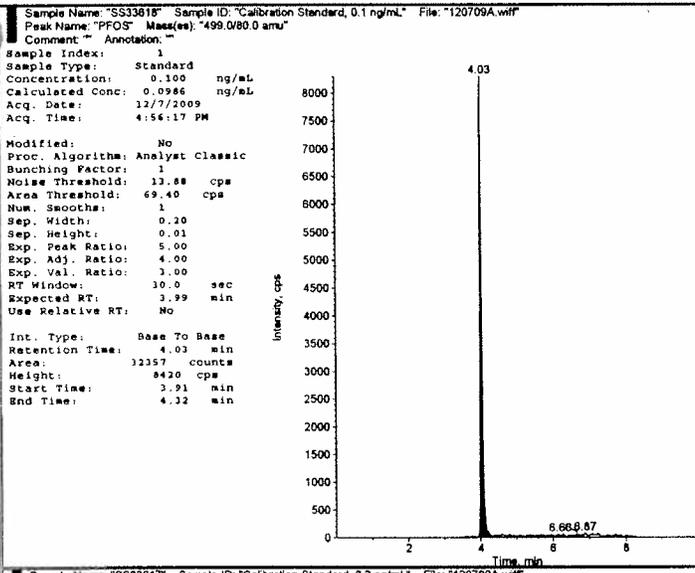
**Analyst:** Mark Neeley *MN 12-7-09*  
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 Phone: (814) 272-1039 FAX: (814) 231-1580

All Handwritten Peak ID's by: *MN 12-8-09*

*MPI 12-8-09*

■ 09\_120709A.rdb (PFOS): "Linear" Regression ("1/x" weighting):  $y = 1.03x + 0.0106$  ( $r = 0.9998$ )





Initials MDN  
Date 12-8-09  
sample index 1 To 15

